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ADDENDUM NO. 6
QUALITY ASSURANCE PROJECT PLAN
SELECTED REVISIONS

FOR THE

BLACKWELL LANDFILL
DUPAGE COUNTY, ILLINOIS

Montgomery Watson File No. 1252008

Prepared For:

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DuPage County, Illinois

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April 1998

MONTGOMERY WATSON

APR 27 1998

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APPROVALS:

U.S. EPA Region V Project Manager	Montgomery Watson QA Officer
U.S. EPA Region V Quality Assurance Reviewer	First Environmental Laboratory QA Officer
Forest Preserve District Project Manager	First Environmental Laboratory Director
Montgomery Watson Project Manager	

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APPENDICES

1.0 INTRODUCTION

A Quality Assurance Project Plan (QAPP) was previously submitted in August 1996 as part of the Pre-Design Investigation Work Plan for the Blackwell Landfill. The QAPP presented the organization, objectives, functional activities, and specific quality assurance (QA) and quality control (QC) procedures for planned sampling and analytical activities. The QAPP also described specific protocols for sampling, sample handling and storage, chain of custody, and laboratory and field analyses.

The following five addenda to the QAPP have been submitted since August 1996:

- Addendum No. 1 was prepared as part of the Expedited Final Design Report for the Leachate Collection System (LCS) (Montgomery Watson, May 1997), and provided additional details for sampling and analysis activities associated with the proposed LCS construction.

- Addendum No. 2 described additional quality assurance and quality control activities associated with construction of Cap Repairs.

- Addendum No. 3 provided an updated list of groundwater monitoring wells to be included in the quarterly groundwater monitoring program.

- Addendum No. 4 was prepared to supplement the August 1996 QAPP and an August 1990 Field Sampling Plan, which addressed sediment sampling. This addendum included updated tables that listed requirements for the analysis of soil samples collected during the North Stormwater Pipe Soil Investigation and water samples collected during surface water sampling of Sand Pond.

- Addendum No. 5 was prepared primarily to include alternative analytical methods that would achieve lower detections limits for some compounds on the groundwater analyte list. The compounds were those for which the analytical method practical quantitation limit (PQL) exceeded the promulgated maximum contaminant level (MCL). In addition, this addendum included a table that was inadvertently omitted from Addendum No. 4, described changes in the instrumentation for measurement of field parameters, and presented an updated organization chart for the Blackwell Landfill Response Action.

This Addendum No. 6 revises Sections 1, 2 and 3 of the August 1996 QAPP based on comments provided in an U.S. EPA letter dated March 23, 1998.

2.0 SELECTED REVISIONS

Information presented in the August 1996 Pre-Design Investigation QAPP and subsequent addenda is applicable except as superseded by this addendum.

U.S. EPA COMMENT NO I: Section 1.6 was not revised according to the previous QAMS comments. DQOs should be based on the seven step process described in EPAQA/G-4 (September 1994) document. Please address the project specific DQOs based on the current US EPA Region 5 Model QAPP (Revision 1, May 1996).

RESPONSE: Section 1.6 of the August 1996 Pre-Design Investigation QAPP is superseded by the following revised text:

1.6 DATA QUALITY OBJECTIVES

The Data Quality Objective (DQO) Process are a series of planning steps based on the Scientific Method that is designed to ensure that the type, quality, and quantity of environmental data used in decision making are appropriate for the intended application.

DQO are qualitative and quantitative statements derived from outputs of each step of the DQO Process that:

- Clarify the study objective;
- Define the most appropriate type of data to collect; and
- Determine the most appropriate conditions from which to collect the data;

The DQO are then used to develop a scientific and resource-effective sampling design.

The DQO Process allows decision makers to define their data requirements and acceptable levels of decision during planning before any data are collected. The DQO process is based on the seven step process described in EPA QA/G-4 (September 1994) document, and is outlined for each task as follows:

1.6.1 Groundwater Monitoring

1. **State the Problem** - Contamination has been detected in the glacial aquifer downgradient from the landfill. Additional routine groundwater sampling, field testing (pH, conductivity, redox potential, dissolved oxygen, turbidity, temperature, carbon dioxide, methane, and oxygen) and laboratory analysis (TCL VOCs and SVOCs, TAL metals/cyanide, chloride, sulfate and TDS) will be required to detect changes in the

chemical composition of the groundwater in both the glacial aquifer and in the underlying bedrock aquifer. These new data will be used to evaluate the site remedy.

2. **Identify the Decision** - Required information to address the problem includes currently available groundwater data and new routine monitoring data which will be used to re-assess the nature and extent of the groundwater plume.

3. **Identify Inputs to the Decision** - Required environmental information includes routine performance of field and laboratory testing of groundwater samples collected from the site monitoring well network.

4. **Define the Study Boundaries** - The study boundaries encompasses the detection and compliance monitoring well network.

5. **Develop a Decision Rule** - Significant changes in the nature and extent of the groundwater plume may require modification to the remedy.

6. **Specify Limits on Decision Errors** - Acceptable limits on decision errors for the laboratory analyses are similar to those used in the RI.

7. **Optimize the Design for Obtaining the Data** - The most resource-efficient sampling and analysis design for generating laboratory data, that are expected to satisfy the DQOs, are similar to those used in the RI. The laboratory analytical data obtained for this task must be validated as specified in Section 9.2. Field testing will satisfy screening requirements.

1.6.2 Soil Analyses

1. **State the Problem** - Areas of the landfill cover may not have a minimum of 2 ft of low permeability material in place. Soil samples will be collected from the cover and analyzed for grain size and permeability to identify deficient areas.

2. **Identify the Decision** - Grain size and permeability data for cover soils are required to assess whether the cover meets material and permeability criteria.

3. **Identify Inputs to the Decision** - New chemical and/or physical data for soil samples are required.

4. **Define the Study Boundaries** - Chemical and/or physical data from soil samples will be obtained from the study area. The study area includes the existing or repaired landfill cover.

5. **Develop a Decision Rule** - Significant variations in physical soil characteristics may require modification of the remedy.

6. **Specify Limits on Decision Errors** - Acceptable limits on decision errors, which are used to establish performance goals for limiting uncertainty in the data, are similar to those in the RI.

7. **Optimize the Design for Obtaining the Data** - The most resource-efficient sampling and analysis design for generating data that are expected to satisfy the DQOs are similar to those used in the RI.

Refer to Table 1-3 for a summary of data generating activities.

U.S. EPA COMMENT NO II: The involvement and responsibility of EPA Quality Assurance Reviewer are not addressed in this Section. Please note, that EPA Superfund Field Services Section (FSS) QA Reviewer is responsible for review and approval of all QAPPs. Please address it in this Section and in Figure 1.

RESPONSE: Section 2.1.1 of the August 1996 Pre-Design Investigation QAPP and Figure 1 are superseded by the following revised text and Figure 1.

2.1.1 U.S. EPA Region V Remedial Project Manager and Quality Assurance Reviewer

The U.S. EPA Region V is the lead agency and is responsible for providing oversight of the Blackwell remedial design. The U.S. EPA Remedial Project Manager has the responsibility for review and approval of this QAPP. The U.S. EPA Remedial Project Manager is Mr. Michael Bellot. His responsibilities encompass acting as the coordinator of communications between the U.S. EPA and FPD/Montgomery Watson, and assuring contract compliance.

The EPA Superfund Field services section (FSS) QA Reviewer is responsible for review and approval of all QAPPs.

U.S. EPA COMMENT NO. III: Section 3.1 needs to be revised: MS/MSD samples are investigative samples. Please correct.

RESPONSE: Section 3.1 of the August 1996 Pre-Design Investigation QAPP, Figure 1 and Figure 3-2 are superseded by the following revised text:

3.1 LEVEL OF QUALITY CONTROL EFFORT

Field blank, trip blank, duplicate, and matrix spike samples will be analyzed to assess the quality of the data resulting from the field sampling program. Field and trip blanks consisting of deionized water, will be submitted to the analytical laboratories to provide the means to assess the quality of the data resulting from the field sampling program. Field blank samples are analyzed to check for procedural contamination at the site which may cause sample contamination. Trip blanks are used to assess the potential for contamination of samples due to contaminant migration during sample shipment and storage. Duplicate samples are analyzed to check for sampling and analytical reproducibility. Matrix spikes are investigative samples that provide information about the effect of the sample matrix on the digestion and measurement methodology. One matrix spike/matrix spike duplicate (MS/MSD) will be collected for every 20 or fewer investigative samples. MS/MSD samples are designated/collected for organic analyses, only. Inorganic analyses require a matrix spike and a duplicate analysis but extra volume is not required.

The general level of the QC effort will be one field duplicate and one field blank for every ten or fewer investigative samples. One VOC trip blank consisting of distilled deionized ultra pure water will be included along with each shipment of aqueous VOC samples.

MS/MSD samples are considered investigative samples. Aqueous MS/MSD samples must be collected at triple the volume for VOCs and double the volume for extractable organics. One MS/MSD sample will be collected/designated for every 20 or fewer investigative samples per sample matrix (i.e., groundwater). The number of duplicate and field blank samples to be collected are listed in Table 1-1. Sampling procedures are specified in the Field Sampling Plan (FSP) (Volume III).

The level of QC effort provided by the laboratory will be equivalent to the level of QC effort specified in Table 3-2.

The QC level of effort for the field measurement of pH consists of pre-measurement calibration and a post-measurement verification using two standard reference solutions each time as appropriate to the sample pH. This procedure will be performed for each sample tested.

The QC effort for field conductivity measurements will include daily premeasurement calibration of the instrument using standard solutions of known conductivity. A check sample of known conductivity will be analyzed after every ten investigative samples. The QC effort for temperature readings will be limited to taking replicate readings.

The QC effort for field dissolved oxygen measurements will include daily calibration of the instrument using an oxygen saturated water solution as a check of the calibration after ten investigative samples.

The QC effort for field measurement of turbidity will include daily calibration of the instrument using a reagent grade water blank and a standard of known turbidity.

The QC effort for field measurement of oxidation/reduction potential will include daily calibration of the instrument using pH buffers of 4 and 7 with quinhydrone crystals and a check of the calibration after ten investigative samples.

The QC effort for field measurement of carbon dioxide, methane, and oxygen will include daily calibration the GEM500 using the appropriate calibration gas cylinders.

U.S. EPA COMMENTS NO. IV THROUGH VI: (Various comments on SOPs)

RESPONSE: Refer to revised Table 3-2 and Appendices A through C for revised SOPs for VOA, SVOA, and ICAP Metals.

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TABLES

TABLE 3-2

Summary of Quality Control Requirements

Pre-Design Investigation - Blackwell Landfill NPL Site
DuPage County, Illinois

PARAMETER	AUDIT	FREQUENCY(1)	LIMITS(2)		
TCL VOCs	Method Blank	Daily	< DL		
	5 point calibration	Start up and as necessary	$R^2 \geq .995$		
	CCV	Every 12 hours	80-120%		
	MS/MSD	1 per 20 samples	Spike Compound	%Rec	%RPD
			1,1-Dichloroethene	61-145	14
			Trichloroethene	71-120	14
			Chlorobenzene	75-130	13
			Toluene	76-125	13
			Benzene	76-125	11
TCL SVOCs	Method Blank	Daily	< DL		
	5 point calibration	Start up and as necessary	$R^2 \geq .995$		
	CCV	Every 12 hours	70-130%		
	MS/MSD	1 per 20 samples	Spike Compound	%Rec	%RPD
			Phenol	12-110	42
			2-Chlorophenol	27-123	40
			1,4-Dichlorobenzene	36-97	28
			N-Nitroso-di-n-propylamine	41-116	38
			1,2,4-Trichlorobenzene	39-98	28
			4-Chloro-3-methylphenol	23-97	42
			Acenaphthene	46-118	31
			2,4-Dinitrotoluene	24-96	38
			4-Nitrophenol	10-80	50
			Pentachlorophenol	9-103	50
			Pyrene	26-127	31
Chloride and sulfate	Lab Blank	After calibration, every 10 samples, and at the end of the run	<Detection Limit		
	Check Standard	After calibration, every 10 samples, and at the end of the run	90-110%Recovery		
	EPA QC Reference Standard	1 per set	80-120 % Recovery		
	Lab Duplicate	1 per 10 samples	10% RPD (+/-2xDL if sample concentration is <5x DL)		
	Matrix Spike	1 per 10 samples	75-125 % Recovery		
Total Dissolved Solids	Lab Blank	1 per set	<Detection Limit		
	EPA QC Reference Standard	1 per set	80-120 % Recovery		
	Lab Duplicate	1 per 10 samples	+/- 20% RPD (+/-2xDL if sample concentration is <5x DL)		

TABLE 3-2

Summary of Quality Control Requirements

Pre-Design Investigation - Blackwell Landfill NPL Site
DuPage County, Illinois

PARAMETER	AUDIT	FREQUENCY(1)	LIMITS(2)
Cyanide	Initial Calibration	Daily	$R^2 \geq 0.995$
	Reagent Blank and CCBs	Daily, beginning, end, and every 10 samples	< DL
	ICS	Immediately following curve	80 - 120 %
	CCS	Daily, beginning, end, and every 10 samples	85 - 115 %
	LCS	1 per 20 samples	80 - 120 %
	Duplicate	1 per 10 samples	+/- 20 % RPD
	MS/MSD	1 per 20 samples	+/- 25 %
TAL Inorganics (ICAP)	Initial Calibration	Daily	Not Applicable
	Calibration Blank	At beginning, end and after every ten samples	<Reporting Limit
	Calibration Check	Daily	+/- 5 %
	Init. Calib. Verif. (ICV)	Beginning of batch	90 - 110%
	Interf. Check Standard	Beginning and end of run batch	80 - 120%
	Lab. Reagent Blank (LRB)	Per batch, where a batch is defined as daily or every 20 samples, whichever is less.	< DL
	Lab. Fortified Blank (LFB)	Per batch, where a batch is defined as daily or every 20 samples, whichever is less.	90 - 110%R
	Duplicate	10%	+/- 20 RPD
	Lab. Fortified Matrix (LFM)	10%	+/- 25 %
	Quality Cont. Sample* (QCS)	Quarterly - APG PE Program	+/- 5 % of mean of three analyses

A LFB and LRB are required only if the sample is digested. If samples are not digested, the Turbidity measurement must be documented as being <1 NTU.
*Also require after preparation of calibration standard solutions.

TABLE 3-2

Summary of Quality Control Requirements

Pre-Design Investigation - Blackwell Landfill NPL Site

DuPage County, Illinois

PARAMETER	AUDIT	FREQUENCY(1)	LIMITS(2)
Mercury	Calibration Check	Daily	90 - 110 %
	Reagent Blank	Daily	< DL
	CCBs	1 per 10 samples	< DL
	LCS	1 per 20 samples	80 - 120 %
	Duplicate	1 per 10 samples	+/- 20 % RPD
	MS/MSD	1 per 20 samples	+/- 25 %
Grain Size	Lab Duplicates	1 per 10 samples	+/- 10 % RPD
Falling Head Permeability	Lab Duplicates	1 per 10 samples	+/- 10 % RPD
pH (Field)	Buffers pH 4 and 7	Daily	+/- 5% of standard
	Check Standard	1 per 10 samples	+/- 5% of standard
	Duplicate	1 per 10 samples	+/- 0.2 pH unit
Dissolved Oxygen (Field)	Oxygen Saturated Std.	1 per 10 samples	+/- 10% from actual
	Duplicate	1 per 10 samples	15% RPD
Specific Conductance (Field)	Check Standard	1 per 10 samples	+/- 10% of standard
	Duplicate	1 per 10 samples	15% RPD
Oxidation/Reduction (Field)	Standards	Daily	+/- 10%
	Check Standard	1 per 10 samples	+/- 10% of standard
	Duplicate	1 per 10 samples	15% RPD
Turbidity (Field)	Standards	Daily	+/- 10%
	Duplicate	1 per 10 samples	15% RPD
Calcium, Magnesium, Potassium , Sodium (Arboreal Investigation)	Standards	Daily	+/- 10%
	Check Standard	1 per 10 samples	+/- 10% of standard
	Duplicate	1 per 10 samples	15% RPD
Soil pH	Buffers pH 4 and 7	Daily	± 5% of standard
	Check Standard	1 per 10 samples	± 5% of standard
	Duplicate	1 per 10 samples	+/- 0.2 pH unit

TABLE 3-2

Summary of Quality Control Requirements

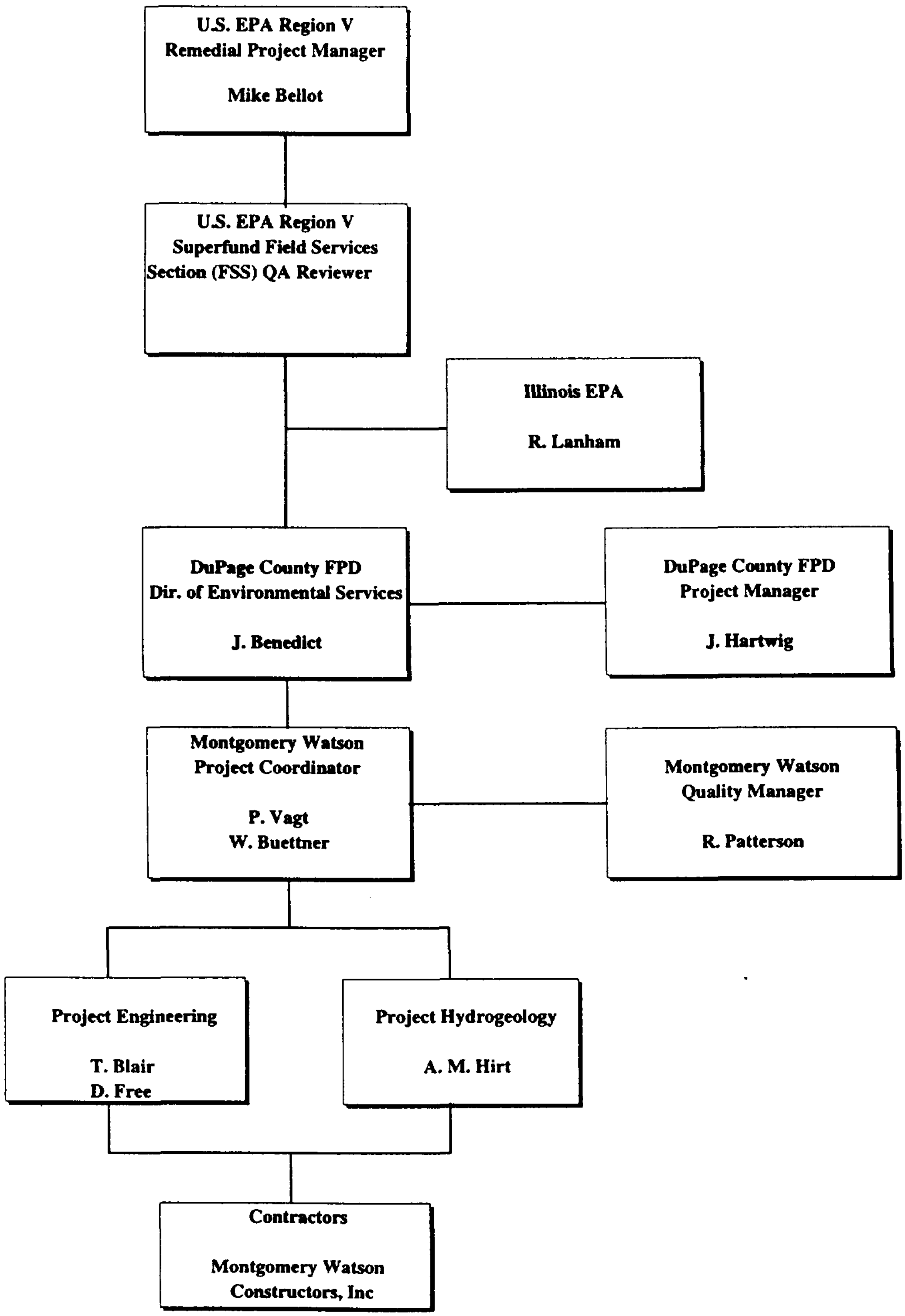
Pre Design Investigation - Blackwell Landfill NPL Site
DuPage County, Illinois

PARAMETER	AUDIT	FREQUENCY(1)	LIMITS(2)
Cation Exchange Capacity	Lab Duplicates	1 per 10 samples	+/- 10 % RPD
Phosphorus	Standards	Daily	+/- 10%
	Check Standard	1 per 10 samples	+/- 10% of standard
	Duplicate	1 per 10 samples	15% RPD

Footnotes:
(1). Frequencies apply to each individual matrix.
(2). Refer to Table 3-1 for required detection limits for each analyte.
(3). QA/QC requirements for analyses of TCLP extract for VOCs, SVOCs, and metals are the same as above.

FIGURE 1

ORGANIZATION CHART
BLACKWELL LANDFILL RESPONSE ACTION



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APPENDIX A

REVISED STANDARD OPERATING PROCEDURE: VOLATILE ORGANICS ANALYSIS; METHOD 8260B

First Environmental Laboratories

Standard Operating Procedure

Title: Volatile Organics Analysis; Method 8260B

Regulatory References: SW-846; 8260B
Regulatory Limits: Varies
Preservation Requirements: Cool, 4°C, HCl (optional)
(Collected without headspace)

Container: 40 mL Vial or 4 oz wide mouth with Teflon lined closure.

Single Analysis Sample Volume: 5.0 mL or ~5g
Holding Time: 7 Days from sample collection.
14 Days if sample is preserved with HCl.

(Range) Reporting Limits (Limits of Detection):

Analyte	Aqueous ug/L	Non-Aqueous ug/kg
Acetone	10.0	10.0
Benzene	5.0	5.0
Bromodichloromethane	5.0	5.0
Bromoform	5.0	5.0
Bromomethane	10.0	10.0
2-Butanone	10.0	10.0
Carbon disulfide	5.0	5.0
Carbon tetrachloride	5.0	5.0
Chlorobenzene	5.0	5.0
Chlorodibromomethane	5.0	5.0
Chloroethane	10.0	10.0
Chloroform	5.0	5.0
Chloromethane	10.0	10.0
1,1-Dichloroethane	5.0	5.0
1,2-Dichloroethane	5.0	5.0
1,1-Dichloroethene	5.0	5.0
1,2-Dichloroethene (total)	5.0	5.0
1,2-Dichloropropane	5.0	5.0
cis-1,3-Dichloropropene	5.0	5.0
trans-1,3-Dichloropropene	5.0	5.0
Ethyl benzene	5.0	5.0
2-Hexanone	10.0	10.0

Analyte	Aqueous ug/L	Non-Aqueous ug/kg
4-Methyl-2-pentanone	10.0	10.0
Methylene chloride	5.0	5.0
Styrene	5.0	5.0
1,1,2,2-Tetrachloroethane	5.0	5.0
Tetrachloroethene	5.0	5.0
Toluene	5.0	5.0
1,1,1-Trichloroethane	5.0	5.0
1,1,2-Trichloroethane	5.0	5.0
Trichloroethene	5.0	5.0
Vinyl Acetate	10.0	10.0
Vinyl Chloride	10.0	10.0
Xylenes (total)	5.0	5.0

Table 1

Summary of Method:

Methods ~~8240B and 8260A~~ 8260B ~~are~~ is used to quantitate most volatile organic compounds that have boiling points below 200°C and that are insoluble or slightly soluble in water. The volatile compounds are introduced into the gas chromatograph by purging the sample aliquot with an inert gas, trapping the components on a sorbent column and finally heating and backflushing the column with helium to desorb the components onto a GC column. The GC is programmed to separate the components, which are detected with a mass spectrometer.

1. Instrumentation / Apparatus / Glassware

For a comprehensive listing of the instrumentation, apparatus and glassware required for this method see "Test Methods for Evaluating Solid Waste", SW-846, Method ~~8240A~~ 8260B Section 4.0.

Analyte-free water used for analysis should be prepared by filling a 500mL Erlenmeyer flask with 400mLs of Laboratory Deionized water and boiling in the microwave oven for 8 minutes. Allow the water to cool while capped under a cold water stream in the VOA analysis room.

The analyst should be familiar with the operation of the Teklink software that controls the Tekmar purge and trap unit, and the Hewlett Packard Enviroquant data acquisition and reduction software before beginning the analysis of samples.

Instrument Conditions:

Aqueous Sample Analysis
Tekmar 3000/2016 Instrument Conditions: (These parameters should be programmed and saved into a Teklink method file named: 8240W.mt3)

Line Temp	120 C
Valve Temp	120 C
MCS Line Temp	120 C
Purge Ready Temp	33 C
Purge Temp	20 C
Purge Time	11 minutes
Purge Flow	38 mL/min
TPC	5 psi
Trap Type	Supelco VOCARB 3000 Type "K"
Desorb Flow	25 mL/min
DryPurge Time	0 minutes
MCS Desorb Temp	50 C
GC Start Option	Start of Desorb
GC Cycle Time	0 minutes
Desorb Preheat Temp	250 C
Desorb Time	4 minutes
Desorb Temp	260 C
Sample Drain	Off
Bake Time	8 minutes
Bake Temp	260 C
Bake Gas Bypass	On
Bake Gas Bypass Delay Time	0.5 minutes
MCS Bake Temp	310 C
20XX Valve Temp	120 C
20XX Line Temp	120 C

Table 2

Non-Aqueous Sample Analysis
Tekmar 3000/2016 Instrument Conditions: (These parameters should be programmed and saved into a Teklink method file named: 8240S.mt3)

Line Temp	120 C
Valve Temp	120 C
MCS Line Temp	120 C
Purge Ready Temp	33 C
Purge Temp	20 C
Sample Heater	On
PrePurge Time	0.0 minutes
Sample Preheat Time	0.5 minutes
Sample Preheat Temp	40 C
Purge Time	11 minutes
Purge Flow	38 mL/min
TPC	5 psi
Trap Type	Supelco VOCARB 3000
Desorb Flow	25 mL/min
DryPurge Time	3 minutes
MCS Desorb Temp	50 C
GC Start Option	Start of Desorb
GC Cycle Time	0 minutes
Desorb Preheat Temp	250 C
Desorb Time	4 minutes
Desorb Temp	260 C
Sample Drain	Off
Bake Time	6 minutes
Bake Temp	270 C
Bake Gas Bypass	On
Bake Gas Bypass Delay Time	0.5 minutes
MCS Bake Temp	310 C
20XX Valve Temp	120 C
20XX Line Temp	120 C

Table 3

GC/MS Instrument Conditions (Split Injection)

In this technique the desorb line from the Tekmar unit is connected to the GC injection port. The sample is introduced to the narrow bore column using the split injection technique. (These parameters should be programmed and saved into a Hewlett Packard Enviroquant method file named: *8240S.M where "*" is the instrument ID letter.) A separate method file should be kept for aqueous and non-aqueous sample analyses. The Hewlett Packard software stores calibration information as well as the analytical parameters in the Method file. Typically, the method name ending with an "S" will be the non-aqueous method file, and the method name ending with a "W" will be the aqueous method file.

Column	HP-624 25meter
Column ID/Thickness	0.2mm ID 1.12um
Interface	Cap Direct Split Inj.
GC Type	HP5890 Series II/Plus
Mass Spectrometer	HP5972A
Tune	BFB Tune + 300v
Scan Range	35-260 amu
Sampling #	3
Threshold	100
Carrier Gas	Helium
Vacuum Comp.	On
Pressure	16psi @ 35 deg C
Constant Flow Mode	1.0 mL/min
Split Flow	25 ml/min
Split Ratio	25:1
Inj. B Temp	220 C
Detector B Temp	280 C
Initial Temp	35 C
Initial Time	4 minutes
Rate(1)	7.00 C/min
Final Temp(1)	120 C
Final Time(1)	0 min
Rate(2)	25.0 C/min
Final Temp(2)	230 C
Final Time(2)	1.50 min
Total GC run time	22.04 min
Purge B	On (during entire run)

Table 4

2. Standards

4-BFB Tuning Solution Preparation

Prepare the 4-BFB solution by diluting 400 uL of Restek Catalog # 4-BFB Mix (2500 ug/mL) to 10.0 mLs in a volumetric flask with "Purge and Trap" Grade methanol.

Store with minimal headspace in 2mL or 5mL vials with mini-nert valves and septa. Store in the VOA freezer at -18 deg C, separate from samples. This solution should be replaced every 6 months.

Internal Standard/System Monitoring Compounds Solution

Prepare the Internal Standard/System Monitoring Compound (SMC) solution by diluting 200 uL of Restek Catalog #30011 VOA Internal Standard Mix (2500 ug/mL each component) and 200 uL of Restek Catalog #30004 VOA LM Surrogate Spike Mix (2500 ug/mL each component) to 10.0 mLs in a volumetric flask with "Purge and Trap" Grade methanol.

Store with minimal headspace in 2mL or 5mL vials with mini-nert valves and septa. Store in the VOA freezer at -18 deg C, separate from samples. This solution should be replaced every 6 months.

Matrix Spike Solution Preparation

Prepare the matrix spike solution by diluting 100 uL of Restek catalog #30005 VOA Matrix Spike Mix (2500 ug/mL each component) to 10.0 mLs in a volumetric flask with "Purge and Trap" Grade methanol.

Store with minimal headspace in 2mL or 5mL vials with mini-nert valves and septa. Store in the VOA freezer at -18 deg C, separate from samples. This solution should be replaced every 6 months.

Initial Calibration Preparation

(8240/8260 Target Compound List)

Prepare the following VOA calibration solutions: Dilute the indicated volumes of each mixture to 10.0 mLs in a volumetric flask using "Purge and Trap Grade" Methanol.

Solution #1:

Catalog Number	Description	Initial Conc	Volume	Final Conc
RK30006	VOA Calibration Mix #1	5000 ug/mL	100 uL	50 ug/mL
RK30007	VOA Calibration Mix #2	2000 ug/mL	250 uL	50 ug/mL
RK30056	8010A Calibration Mix #2	2000 ug/mL	250 uL	50 ug/mL

RK30057	8010A Calibration Mix #3	2000 ug/mL	250 uL	50 ug/mL
RK30077	8240 Volatiles Mix #1	2000 ug/mL	250 uL	50 ug/mL
RK30078	8240 Volatiles Mix #2	2000 ug/mL	250 uL	50 ug/mL

Table 5

Store with minimal headspace in 2mL or 5mL vials with mini-nert valves and septa. Store in the VOA freezer at -18 deg C, separate from samples. This solution should be replaced every 6 months.

The gases mixture, RK30042, is prepared in a separate solution. The gases have a tendency to degrade/evaporate faster than the other compounds and this solution should be replaced monthly.

Solution #2:

Catalog Number	Description	Initial Conc	Volume	Final Conc
RK30042	502.2 Calibration Mix #1	2000 ug/mL	250 uL	50 ug/mL

Store with minimal headspace in 2mL or 5mL vials with mini-nert valves and septa. Store in the VOA freezer separate from samples.

Prepare the 5-point calibration using the following amounts of the calibration solutions added to 5.0mLs of DI water along with the appropriate amounts of internal standard and surrogate spiking solutions:

Std Conc	5.0 ppB	10.0 ppB	20.0 ppB	50.0 ppB	100 ppB	200 ppB
Amount	0.5 uL	1.0 uL	2.0 uL	5.0 uL	10.0 uL	20.0 uL

Analyze the initial calibration curve according to the analysis procedure in Section 4.

Tabulate the table of response factors (RF) for each compound. The RF is calculated as follows:

$$RF = (A_x C_{is}) / (A_{is} C_x)$$

where:

A_x	=	Area of characteristic ion for the compound being measured.
A_{is}	=	Area of the characteristic ion for the specific internal standard.
C_{is}	=	Concentration of the specific internal standard.
C_x	=	Concentration of the compound being measured.

The RF, the average RF and the %RSD is automatically calculated with the data system software. The initial calibration response factor report should be printed and included with the daily QC file folder. The 5 SPCC compounds should be evaluated and meet the minimum response factors listed in Table 6. The %RSD for each of the CCC compounds listed in Table 6 must be less than 30%.

Compound	Minimum RF Criteria	Initial Calib Max %RSD	Cont. Calib Max %RSD
Bromoform	>0.10		
Chlorobenzene	0.30		
Chloroform		30	20
Chloromethane	0.10		
1,1-Dichloroethane	0.10		
1,1-Dichloroethene		30	20
1,2-Dichloropropane		30	20
Ethylbenzene		30	20
1,1,2,2-Tetrachloroethane	0.30		
Toluene		30	20
Vinyl chloride		30	20

Table 6

3. Safety

The analyst is required to fully read and understand the Safety Standard Operating Procedure.

Each analyst is directly responsible for complete understanding of all health hazards associated with every chemical and procedure that is used. The analyst must be aware of these hazards and all protective wear and clean-up procedures prior to the use of any chemical. In all cases, both the applicable MSDS and Safety Officer should be consulted. Safety information that is indicated on each chemical container must be read. Protective gloves and safety glasses must be worn, and solvents will be handled in ventilated hoods, in addition to the safety measures that are dictated in the Safety SOP.

4. Analytical Procedures

4.1 Mass Spectrometer Tuning

Tune the GC/MS system using "Target Tune". Store the tune values in the file named "bfb.u". Create a daily QC results folder and store the hardcopy output from this tune in that file.

☞ Look at the spectra from the Tune report. Ion 131 should be higher in abundance than ion 219. Both of these relative abundances should be around 35%. If it is not, repeat the tune steps listed above.

4.2. **Analyze Tuning Compound 4-BFB:** Inject 2.0 uL of 4-BFB tuning solution (25 ng/uL) into the gas chromatograph injection port. When setting up your batch sequence, indicate that this run is "BFB" in the sample type field. This will automatically evaluate the BFB run and issue a report. In this report, all ions should indicate "PASS". If not, re-tune as in 4.1 above and repeat the 4-BFB analysis. The mass spectrum of the 4-BFB must meet the following criteria:

Mass	Intensity Required (relative abundance)
50	15 to 40% of mass 95
75	30 to 60% of mass 95
95	base peak, 100% relative abundance
96	5 to 9% of mass 95
173	less than 2% of mass 174
174	greater than 50% of mass 95
175	5 to 9% of mass 174
176	greater than 95% but less than 101% of mass 174
177	5 to 9% of mass 176

Table 6

4.3 **Aqueous Analysis: Analyze Daily Continuing Calibration Standard:** Remove the plunger from a 5mL gas-tight syringe. Fill the syringe with Boiled DI water and replace the syringe plunger. Open the syringe valve and adjust the volume to 5.0mLs. Add 5.0 uL each of the VOA standard solution (Solution #1 in Section 2) and VOA gases mixture (Solution #2) to the syringe. Add 5.0 uL of Internal Standard/SMC solution. Load this sample into a clean position on the Tekmar Purge and Trap autosampler. Purge this sample using the conditions and method parameters detailed in section 1.

To evaluate the results of this run enter the ENVDA program. Select "File" and load the correct data file. Select "Method" and load the proper method, 8240'V.M. Select the option "Concal", then select "Evaluate Data as Continuing Calibration to Printer". This will generate a report that compares this standard analysis to the initial calibration curve. The CCC compounds (as detailed in Table 6) should all be less than 20% drift (Compounds above 20% will be indicated with a "#" on this report). If the specific CCC compounds are less than 20%, the initial calibration is assumed to be valid. If the criterion is not met, corrective action must be taken. If the source of the problem cannot be determined after corrective action has been taken, a new 5-point calibration must be generated.

4.4 **Analyze Daily Method Blank**: Remove the plunger from a 5mL gas-tight syringe. Fill the syringe with Boiled DI water and replace the syringe plunger. Open the syringe valve and adjust the volume to 5.0mLs. Add 5.0 uL ISTD/SMC solution to 5.0 mLs boiled DI water. Load this sample into a clean position on the Tekmar Purge and Trap autosampler. Purge this sample using the conditions and method parameters detailed in section 1.

4.5 **Evaluate the Method Blank**. No target compounds should be present. ~~Methylene chloride must be below 25 ug/L and Acetone <50 ug/L (Unless you are analyzing for BTEX only).~~ Proceed with sample analysis if all indicators are within control limits.

4.6 **Analyze Samples**. Remove the plunger from a 5mL gas-tight syringe. Fill the syringe with the sample to be analyzed and replace the syringe plunger. Open the syringe valve and adjust the volume to 5.0mLs. Add 5.0 uL ISTD/SMC solution to 5.0 mLs boiled DI water. Load this sample into a clean position on the Tekmar Purge and Trap autosampler. Purge this sample using the conditions and method parameters detailed in section 1.

4.7 **Soil Analysis:** Analyze the Daily Continuing Calibration standard: Analyze this sample the same as in step 4.1 above. Purge this sample (after covering the tube with a pocket heater) using the conditions and method parameters detailed in section 1 for soil analysis. Evaluate the results of this run as in step 4.3 above.

4.8.1 **Analyze Daily Method Blank**: Weigh approximately 2 to 5 grams of clean sand into a purge tube. Add 5.0 uL ISTD/SMC solution to 5.0 mLs boiled DI water. Purge this sample using the conditions and method parameters detailed in section 1.
4.8.2 **Evaluate the Method Blank**. No target compounds should be present. ~~Methylene chloride must be below 25 ug/kg and Acetone <50 ug/kg (Unless you are analyzing for BTEX only).~~ Proceed with sample analysis if all indicators are within control limits.

4.9 **Analyze each sample** by adding 5.0 uL ISTD/SMC solution in 5mLs of DI water to approximately 2 to 5 grams of sample in a purge tube. Purge this sample (after covering the tube with a pocket heater) using the conditions and method parameters detailed in section 1 for soil analysis. Evaluate the results of this run as in step 4.3 above.

4.9.1 **Sample Calculations.** Calculate the concentration of each identified analyte in the sample as follows:

Water Concentration (ug/L) = $(A_x)(I_s) / (A_{is})(RF)(V_o)$

where:

A_x	=	Area of characteristic ion for compound being measured
I_s	=	Amount of internal standard being injected (ng).
A_{is}	=	Area of characteristic ion for the internal standard
RF	=	Mean relative response factor for compound being measured.
V_o	=	Volume of water purged (mL), taking into consideration any dilutions made.

Soil Concentration (ug/kg) = $(A_x)(I_s)(V_t)$ / $(A_{is})(RF)(V_i)(W_s)(D)$

where:

A_x	=	Area of characteristic ion for compound being measured
I_s	=	Amount of internal standard being injected (ng).
A_{is}	=	Area of characteristic ion for the internal standard
RF	=	Mean relative response factor for compound being measured.
V_t	=	Volume of total extract (uL) (Use 10,000 uL or a factor of this when dilutions are made)
V_i	=	Volume of extract added for purging.
W_s	=	Weight of sample extracted or purged (g).
D	=	% dry weight of sample/100, or 1 if sample is to be reported on a wet-weight basis.

4.10 **MS/MSD**. Analyze a MS/MSD pair for every 20 sample analyses or according to the frequency dictated by the QAPP for the sample project. The MS/MSD spike is prepared by adding 10uLs of the spiking solution to each sample and analyzing the sample using the same conditions as the rest of the sample batch. The following sub-set compounds should be used. Recovery criteria will be established and updated by determining the standard deviation every 30 samples. The UCL and LCL will be +/- 3 times the standard deviation. Control charts with this data will be maintained and updated on a routine basis. Acceptable spike recovery and %RPDs must fall within the limits in the following table:

Spike Compound	% Rec Limits	% RPD
1,1-Dichloroethene	61-145	14
Trichloroethene	71-120	14
Chlorobenzene	75-130	13
Toluene	76-125	13
Benzene	76-127	11

Table 7

5.0 Interferences

The analytical system should be checked to ensure freedom from interferences, under the analysis conditions, by analyzing method blanks. Cross-contamination can occur whenever high-concentration and low-concentration samples are analyzed sequentially. Whenever an unusually concentrated sample is analyzed, it should be followed by the analysis of organic-free reagent water to check for carry-over contamination. The low-concentration sample that may have followed a high-concentration sample should be re-analyzed to confirm the analytical result.

Special precautions should be taken to analyze for methylene chloride. The analytical and sample storage area should be isolated from all atmospheric sources of methylene chloride, otherwise random background levels will result. Laboratory clothing worn by the analyst should be clean since clothing previously exposed to methylene chloride fumes during liquid/liquid extraction procedures can contribute to sample contamination.

A comprehensive listing of possible interferences can be found in SW-846 Method ~~8240B~~ 8260B Section 3.0~~ and also in Method 8260A Section 3.0~~. The analyst must read these sections prior to performing analyses.

6. Additional Quality Control Indicator Assessments

The internal standard responses and retention times in the analytical batch must be evaluated immediately after or during data acquisition. If the retention time for any internal standard changes by more than 30 seconds from the last calibration check (12 hours), the chromatographic system must be inspected for malfunctions and corrections must be made as required. If the EICP area for any one of the internal standards changes by a factor of two (-50% to +100%) from the last daily calibration check standard, the mass spectrometer must be inspected for malfunctions and corrections must be made as appropriate. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is required.

Surrogate compounds will be added to every standard, blank and sample that is analyzed. The surrogate recoveries must fall within the following speficiations. If any one recovery is outside these limits, the sample must be re-analyzed to establish that a sample matrix effect is present.

Surrogate Compound	**Low/High Water**	**Low/High Soil/Sediment**
4-Bromofluorobenzene	86 - 115	74 - 121
1,2-Dichloroethane-d_4	76 - 114	70 - 121
Toluene-d_8	88 - 110	81 - 117

7. Notes

Routine Maintenance. The analyst should be familiar with the routine maintenance procedures for both the chromatographic and mass spectrometer systems. Routine maintenance should include, but not be limited to, septum changes, absorbent trap replacement, mass spectrometer source cleaning, vacuum pump fluid replacement and purge and trap system decontamination procedures. The analyst must be aware of system indicators and quality control indicators that warrant attention to these maintenance procedures. The analyst should contact a supervisor if there is any question about these procedures.

8. Approvals

Reviewed for Technical Accuracy by: Neal E Clephon

Reviewed for Quality Assurance Compliance by: L. Franklin

Implementation Date: 04/15/98

End Use Date: ____________________

APPENDIX B

REVISED STANDARD OPERATING PROCEDURE:
SEMI-VOLATILE ORGANICS ANALYSIS;
METHOD 8270C BASE-NEUTRAL AND ACID (BNA) ANALYSIS

First Environmental Laboratories

Standard Operating Procedure

Title: Semi-Volatile Organics Analysis; Method ~~8270B~~ 8270C
Base-Neutral and Acid (BNA) Analysis

Regulatory References: SW-846

Regulatory Limits: Varies

Preservation Requirements: Cool, 4°C

Container: One quart amber or 16 oz wide mouth with Teflon lined closure.

Single Analysis Sample Volume: 1000mL or ~30g.

Holding Time: 7 Days from sample collection (aqueous).
14 Days from sample collection (non-aqueous).
Sample extracts must be analyzed within 40 days from the date of extraction.

(Range) Reporting Limit: 10 to 50 ug/L (ground water samples)
330 to 1650 ug/kg (soil/sediment samples).
Sample reporting limits are highly matrix dependent and will be proportionally higher for sample extracts that require dilution to avoid saturation of the detector.

	Aqueous ug/L	Non- Aqueous ug/kg
Acenaphthene	10	330
Acenaphthylene	10	330
Anthracene	10	330
Benzidine	10	330
Benzo[a]anthracene	10	330
Benzo[b]fluoranthene	10	330
Benzo[k]fluoranthene	10	330
Benzo[g,h,i]perylene	10	330
Benzo[a]pyrene	10	330
Benzoic Acid	50	330
Benzyl alcohol	20	330
bis(2-Chloroethoxy)methane	10	330
bis(2-Chloroethyl)ether	10	330
bis(2-chloroisopropyl)ether	10	330

	Aqueous ug/L	Non- Aqueous ug/kg
bis(2-Ethylhexyl)phthalate	10	330
4-Bromophenyl-phenylether	10	330
Butylbenzylphthalate	10	330
4-Chloroaniline	20	1600
4-Chloro-3-methylphenol	20	1600
2-Chloronaphthalene	10	330
2-Chlorophenol	10	330
4-Chlorophenyl-phenylether	10	330
Chrysene	10	330
Dibenz[a,h]anthracene	10	330
Dibenzofuran	10	330
1,2-Dichlorobenzene	10	330
1,3-Dichlorobenzene	10	330
1,4-Dichlorobenzene	10	330
3,3'-Dichlorobenzidine	20	660
2,4-Dichlorophenol	10	330
Diethylphthalate	10	330
2,4-Dimethylphenol	10	330
Dimethylphthalate	10	330
Di-n-butylphthalate	10	330
4,6-Dinitro-2-methylphenol	50	1600
2,4-Dinitrophenol	50	1600
2,4-Dinitrotoluene	10	330
2,6-Dinitrotoluene	10	330
Di-n-octylphthalate	10	330
Fluoranthene	10	330
Fluorene	10	330
Hexachlorobenzene	10	330
Hexachlorobutadiene	10	330
Hexachlorocyclopentadiene	10	330
Hexachloroethane	10	330
Indeno[1,2,3-cd]pyrene	10	330
Isophorone	10	330
2-Methylnaphthalene	10	330
2-Methylphenol	10	330
4-Methylphenol	10	330
Naphthalene	10	330
2-Nitroaniline	50	1600
3-Nitroaniline	50	1600
4-Nitroaniline	20	1600
Nitrobenzene	10	330
2-Nitrophenol	10	1600
4-Nitrophenol	50	1600
N-Nitrosodimethylamine	10	330
N-Nitroso-di-n-propylamine	10	330

	Aqueous ug/L	Non- Aqueous ug/kg
n-Nitrosodiphenylamine	10	330
Pentachlorophenol	50	1600
Phenanthrene	10	330
Phenol	10	330
Pyrene	10	330
1,2,4-Trichlorobenzene	10	330
2,4,5-Trichlorophenol	10	660
2,4,6-Trichlorophenol	10	330

Summary of Method:

Method ~~8270B~~ 8270C is used to quantitate most neutral, acidic and basic compounds that are soluble in methylene chloride and capable of being eluted without derivatization as sharp peaks from a gas chromatographic fused-silica capillary column coated with a slightly polar silicone. The GC is programmed to separate the components, which are detected with a mass spectrometer. Prior to using this method, the samples should be prepared by using the appropriate sample preparation and clean-up methods.

1. Instrumentation / Apparatus / Glassware

For a comprehensive listing of the instrumentation, apparatus and glassware required for this method see "Test Methods for Evaluating Solid Waste", SW-846, Method ~~8270B~~ 8270C Section 4.0.

The analyst should be familiar with the operation of the Hewlett Packard "Enviroquant" data acquisition and reduction software before beginning the analysis of samples.

Analyte free water is obtained from the Laboratory's deionized water system.

GC/MS Instrument Conditions (Splitless Injection)

In this technique the electronically controlled injection port pressure is programmed to allow the maximum amount of analyte to be introduced to the analytical column. The sample is introduced to the narrow bore column using the splitless injection technique.

Parameter	Setting
Column	HP-5MS 30meter or Restek XTI-5 30meter
Column ID/Thickness	0.25mm ID 1.12um
Interface	Cap Direct Splitless Inj.
GC Type	HP5890 Series II/Plus
Mass Spectrometer	HP5972A

Parameter	Setting
Tune	DFTPP Tune
Scan Range	35-510 amu
Sampling #	3
Threshold	100
Carrier Gas	Helium
Vacuum Comp.	On
Pressure(1)	20psi @ 50 deg C
Time Pressure(1)	1.0 min
Pressure Rate(1)	99 psi/min
Pressure(2)	5.8 psi
Constant Flow Mode	0.84 mL/min
Inj. B Temp	250 C
Detector B Temp	280 C
Initial Temp	50 C
Initial Time	3 minutes
Rate(1)	10.00 deg C/min
Final Temp(1)	200 deg C
Final Time(1)	0 min
Rate(2)	20.0 deg C/min
Final Temp(2)	290 deg C
Final Time(2)	10.0 min
Total GC run time	32.50 min
Purge B	Off (for 1.0 min)

Table 1

2. Standards

DFTPP System Check Standard

The DFTPP system check standard is purchased prepared from Supelco (Catalog #4-7387). This solution contains DFTPP, 4,4'-DDT, Pentachlorophenol and Benzidine at 50 ug/mL of each component.

Surrogate Spike:

Prepare individual surrogate spiking solutions for Base/Neutral and Acid fractions.

Dilute 1.0mL of Acid or Base/Neutral Surrogate Standard Mix to 50.0 mL with Methanol and place into a clean 4 ounce amber jar with a teflon closure. This solution should be stored at 4°C. BNA samples should be spiked with 1.0mL of each solution

Acid Surrogate Standard Mix:	Restek catalog #31087;	10,000ug/mL each component, 5mL Vial.
Acid Surrogate Standard Mix:	Restek catalog #31063;	10,000ug/mL each component, 1mL Vial.
B/N Surrogate Standard Mix:	Restek catalog #31062;	5,000ug/mL each component, 1mL Vial.
B/N Surrogate Standard Mix:	Restek catalog #31086;	5,000ug/mL each component, 5mL Vial.

MS/MSD Spike:

Prepare a combined solution of the B/N and Acid spiking compounds. Dilute 1.0mL each of the B/N Matrix Spike Mix (Restek #31074) and Acid Matrix Spike Mix (Restek #31061) to 50.0mLs in Methanol. BNA samples should be spiked with 1.0mL of this solution.

Internal Standard Solution

The internal standard solution for BNA extracts is prepared first by sonicating the ISTD mix (RK#31006 which contains 4000 ug/mL of each ISTD compound) for 10 minutes. If particulates still appear in the bottom of the ISTD ampule, sonicate the solution longer until these particles go into solution. Then dilute 1.0mL of the sonicated ISTD mix to a 2.5mL final volume. This is your spiking solution. Prior to spiking samples or standards, sonicate this solution for 10 minutes. Then spike each 1.0mL of BNA extract with 25.0uLs of this solution. This will result in an extract concentration of 40 ug/mL for each individual ISTD component.

Semi-Volatile Initial Calibration Preparation

Prepare the following mother solutions by diluting the indicated volumes to 10.0mLs in a volumetric flask:

Semi-Volatile Mother Solution #1

Calibration Mixture	Catalog #	Initial Concentration	Volume
SV Calibration Mix #1 Anilines	RK31007	2000 ug/mL	1.0mL
SV Calibration Mix #4 Nitroaromatics	RK31010	2000	1.0
SV Calibration Mix #7 Dichlorobenzene	RK31013	2000	1.0
Method 605 Benzidines Mix	RK31030	2000	1.0
B/N Surrogate Std Mix	RK31024	1000	1.0

Table 2

Semi-Volatile Mother Solution #2

Calibration Mixture	Catalog #	Initial Concentration	Volume
SV Calibration Mix #2 Phenols	RK31008	2000 ug/mL	1.0mL
SV Calibration Mix #3 Ethers/phthal.	RK31009	2000	1.0
SV Calibration Mix #5 PNAs	RK31011	2000	1.0
Acid Surrogate Std Mix	RK31025	2000	1.0
Pyridine Std Soln	4-8305	2000	1.0

Table 3

Prepare the 5 point calibration curve using the 2 mother solutions according to the following (these may be prepared in silanized 1.5mL amber vials):

Calibration Solution	20 ug/mL	50 ug/mL	80 ug/mL	100 ug/mL	120 ug/mL	160 ug/mL
Mother Soln #1	50 uL	125 uL	200 uL	250 uL	300	400
Mother Soln #2	50	125	200	250	300	400
uLs Methylene Chloride	900	750	600	500	400	200

Table 4

Prior to analysis add the appropriate amount of ISTD solution to each vial. (25uL of a 1600 ug/mL solution will yield 40 ug/mL). To conserve standard solutions, you may use exactly half of the amounts indicated in table 4 above. (Use half of the routine ISTD solution amount.)

This is summarized in Table 5 below:

Calibration Solution	20 ug/mL	50 ug/mL	80 ug/mL	100 ug/mL	120 ug/mL	160 ug/mL
Mother Soln #1	25 uL	62.5 uL	100 uL	125	150	200
Mother Soln #2	25	62.5	100	125	150	200
uLs Methylene Chloride	450	375	300	250	200	100

Table 5

Analyze the initial calibration curve according to the analysis procedure in Section 4.

Tabulate the table of response factors (RF) for each compound. The RF is calculated as follows:

$$RF = (A_x C_{is}) / (A_{is} C_x)$$

where:

A_x = Area of characteristic ion for the compound being measured.
A_{is} = Area of the characteristic ion for the specific internal standard.
C_{is} = Concentration of the specific internal standard.
C_x = Concentration of the compound being measured.

The RF, the average RF and the %RSD is automatically calculated with the data system software. The initial calibration response factor report should be printed and included with the daily QC file folder. The 5 SPCC compounds should be evaluated and meet the minimum response factors listed in Table 6. The %RSD for each of the CCC compounds listed in Table 6 must be less than 30% in the intial calibration.

Compound	Minimum RF Criteria	Initial Calib Max %RSD	Cont. Calib Max %RSD
N-niroso-di-n-propylamine	0.05		

Compound	Minimum RF Criteria	Initial Calib Max %RSD	Cont. Calib Max %RSD
Hexachlorocyclopentadiene	0.05		
2,4-Dinitrophenol	0.05		
4-Nitrophenol	0.05		
Acenapthene		30	20
1,4-Dichlorobenzene		30	20
Hexachlorobutadiene		30	20
N-Nitrosodiphenylamine		30	20
Di-n-octylphthalate		30	20
Fluoranthene		30	20
Benzo(a)pyrene		30	20
4-Chloro-3-methylphenol		30	20
2,4-Dichlorophenol		30	20
2-Nitrophenol		30	20
Phenol		30	20
Pentachlorophenol		30	20
2,4,6-Trichlorophenol		30	20

Table 6

3. Safety

The analyst is required to fully read and understand the Safety Standard Operating Procedure.

Each analyst is directly responsible for complete understanding of all health hazards associated with every chemical and procedure that is used. The analyst must be aware of these hazards and all protective wear and clean-up procedures prior to the use of any chemical. In all cases, both the applicable MSDS and Safety Officer should be consulted. Safety information that is indicated on each chemical container must be read. Protective gloves and safety glasses must be worn, and solvents will be handled in ventilated hoods, in addition to the safety measures that are dictated in the Safety SOP.

4. Analytical Procedures

4.1 **Tune the GC/MS system** using "Target Tune". Store the tune values in the file named "dftpp.u". Store the hardcopy output in the daily QA/QC file folder.

4.2. **Analyze Tuning Compound DFTPP**: Prior to the analysis of any standards or samples, a DFTPP must be analyzed and meet quality criteria. This must be done at the start of every 12-hour batch. Inject 1.0 uL of DFTPP tuning solution (25 ng/uL) into the gas chromatograph injection port. When setting up your batch sequence, indicate that this run is "DFTPP" in the sample type field. This will automatically evaluate the DFTPP run and issue a report. In this report, all ions should indicate "PASS". If not, re-tune as in 4.1 above and repeat the DFTPP analysis. The mass spectrum of the DFTPP must meet the following criteria:

Mass	Intensity Required (relative abundance)
51	30 to 60% of mass 198
68	<2% of mass 69
70	<2% of mass 69
127	40 to 60% of mass 198
197	<1% of mass 198
198	Base peak, 100% relative abundance
199	5 to 9% of mass 198
275	10 to 30% of mass 198
365	>1% of mass 198
441	Present but less than mass 443
442	> 40% of mass 198
443	17 to 23% of mass 442

Table 7

4.3 **Analyze Initial Calibration Standards.** Analyze the 5-point calibration mixtures that are detailed in Section 2; Table 4 or 5. Analyze these samples using the conditions and method parameters detailed in Section 1.

4.4 The percent relative deviation (%RSD) should be less than 15% for all compounds. The %RSD for each CCC compound (Table 4 from method ~~8270B~~8270C) must be less than 30%. The relative retention times of each compound in each calibration run should agree within 0.06 relative retention time units.

4.5 **Analyze Daily Continuing Calibration Standard**: Prepare and analyze the 50.0 ppm concentration standard. Analyze this sample using the conditions and method parameters detailed in section 1.

To evaluate the results of this run enter the ENVDA program. Select "File" and load the correct data file. Select "Method" and load the proper method, 8270a.m. Select the option "Concal", then select "Evaluate Data as Continuing Calibration to Printer". This will generate a report that compares this standard analysis to the initial calibration curve. All of the continuing calibration compounds should all be less than 20% (Compounds above 20% will be indicated with a "#" on this report). If any of these are out of control, re-run the standard or prepare a new 5-point calibration. (Check the areas of the peaks with QEDIT to be sure they have been integrated properly) A Continuing Calibration Standard must be analyzed every 12 hours during analysis.

4.6 **Analyze Extraction Method Blank**: Analyze this sample using the conditions and method parameters detailed in section 1.

Evaluate the Method Blank. No target compounds should be present (with the exception of phthalate compounds which should not be present above 5 times the PQL). Proceed with sample analysis if all indicators are within control limits.

4.7 **Analyze each sample.** Analyze each standard, blank, MS, MSD, LCS and sample using the conditions and method parameters detailed in section 1. Evaluate each sample chromatogram for the presence/absence of target compound analytes.

4.7.1 **Sample Calculations.** Calculate the concentration of each identified analyte in the sample as follows:

Water Concentration (ug/L) = $(A_x)(I_s)(V_{ex}) / (A_{is})(RF)(V_o)$

where:

A_x	=	Area of characteristic ion for compound being measured
I_s	=	Amount of internal standard being injected (ng).
A_{is}	=	Area of characteristic ion for the internal standard
RF	=	Mean relative response factor for compound being measured.
V_o	=	Volume of liquid extracted taking into consideration any dilutions made.
V_{ex}	=	Volume of final extract (mL)

Soil Concentration (ug/kg) = $(A_x)(I_s)(V_t) / (A_{is})(RF)(W_s)(D)$

where:

A_x	=	Area of characteristic ion for compound being measured
I_s	=	Amount of internal standard being injected (ng).
A_{is}	=	Area of characteristic ion for the internal standard
RF	=	Mean relative response factor for compound being measured.
V_t	=	Volume of final extract (uL)
W_s	=	Weight of sample extracted (g).
D	=	% dry weight of sample/100, or 1 if sample is to be reported on a wet-weight basis.

4.8 **Analyze a MS/MSD** pair for every 20 sample analyses or according to the frequency dictated by the QAPP for the sample project.. The MS/MSD pair should be evaluated for accuracy (% recovery) and precision (%RPD). The results should be tabulated and included in the daily QC package. The following sub-set compounds should be used. Recovery criteria will be established and updated by determining the standard deviation every 30 samples. The UCL and LCL will be +/- 3 times the standard deviation. Control charts with this data will be maintained and updated on a routine basis.

Spike Compound	% Rec Limits	% RPD
Phenol	12 - 110	42
2-Chlorophenol	27 - 123	40
1,4-Dichlorobenzene	36 - 97	28
N-Nitroso-di-n-propylamine	41 - 116	38
1,2,4-Trichlorobenzene	39 - 98	28
4-Chloro-3-methylphenol	23 - 97	42
Acenaphthene	46 - 118	31
2,4-Dinitrotoluene	24 - 96	38
4-Nitrophenol	10 - 80	50
Pentachlorophenol	9 - 103	50
Pyrene	26 - 127	31

Table 8

5. Interferences

The analytical system should be checked to ensure freedom from interferences, under the analysis conditions, by analyzing method blanks. Cross-contamination can occur whenever high-concentration and low-concentration samples are analyzed sequentially. Whenever an unusually concentrated sample is analyzed, it should be followed by the analysis of solvent to check for carry-over contamination. The low-concentration sample that may have followed a high-concentration sample should be re-analyzed to confirm the analytical result. To reduce carry-over, the sample syringe should be rinsed out between sample injections.

6. Quality Control Indicator Assessment

DFTPP and a continuing calibration standard must be run every 12 hours prior to the analysis of samples.

A method blank should be analyzed with every analytical batch. No target compounds should be present ~~(with the exception of phthalate compounds which should not be present above 5 times the PQL).~~.

An MS/MSD pair should be extracted for every twenty samples.

A Laboratory Control Spike (LCS) may be required if the MS/MSD is outside control limits. An LCS should be extracted for every 20 samples in the event it will need to be evaluated. An LCS is an aliquot of laboratory DI water spiked with the MS/MSD spiking solution.

The internal standard responses and retention times in the analytical batch must be evaluated immediately after or during data acquisition. If the retention time for any internal standard changes by more than 30 seconds from the last calibration check (12 hours), the chromatographic system must be inspected for malfunctions and corrections must be made as required. If the EICP area for any one of the internal standards changes by a factor of two (-50% to +100%) from the last daily calibration check standard, the mass spectrometer must be inspected for malfunctions and corrections must be made as appropriate. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is required.

Surrogate compounds will be added to every standard, blank and sample that is analyzed. The surrogate recoveries must fall within the following speficiations. If any one recovery is outside these limits, the sample must be re-analyzed to establish that a sample matrix effect is present.

Surrogate Compound	Low/High Water	Low/High Soil/Sediment
Nitrobenzene-d5	35-114	23-120
2-Fluorobiphenyl	43-116	30-115
Terphenyl-d14	33-141	18-137
Phenol-d5	10-110	24-113
2-Fluorphenol	21-110	25-121
2,4,6-Tribromophenol	10-123	19-122

Table 9

7. Notes

Routine Maintenance. The analyst should be familiar with the routine maintenance procedures for both the chromatographic and mass spectrometer systems. Routine maintenance should include, but not be limited to, septum changes, column cutting, mass spectrometer source cleaning, vacuum pump fluid replacement and GC inlet cleaning. The analyst must be aware of system indicators and quality control indicators that warrant attention to these maintenance procedures. The analyst should contact a supervisor if there is any question about these procedures.

8. Approvals

Reviewed for Technical Accuracy by: Neal E Olsson

Reviewed for Quality Assurance Compliance by: S. Franklin

Implementation Date: 04/15/98

End Use Date: ____________

APPENDIX C

REVISED STANDARD OPERATING PROCEDURE:
ICAP METALS

File name: \\madre\company\word files\sop\blackwel\icpdw.doc
Date: 2/3/96
Page 1 of 1

First Environmental Laboratories

Standard Operating Procedure

Title: ICP Metals

Regulatory References: SW-846 Method 6010A

Preservation Requirements: HNO_3 ph <2. All samples should be held at 4°C until analysis. Preservation is comple after the acidified sample has been held for 16 hours. Before sample processing is started, sample pH must be verified to be less than 2.

Container: 250 or 500 cc plastic or 16 oz jar (use 500cc plastic if Hg analysis is also required)

Single Analysis Sample Volume: 100 mL / 1 g

Holding Time: 6 months

(Range) Reporting Limit - Upper End: analyte specific

Summary of Method: If necessary, samples are digested prior to analysis (see prep bench reference). Samples having a turbidity >1 NTU must be digested. If samples are not digested, the tubidity must be documented as being <1 NTU. Samples are nebulized and the resulting aerosol is transported to the plasma torch. Element specific emission spectra are produced by a radio-frequency inductively coupled plasma. The spectra are dispersed by a grating spectrometer, and the intensities of the line spectra are monitored at specific wavelengths by photomultipliers. Photocurrents from the photomultipliers are processed and controlled by a computer system. Background correction is required to compensate for variable background contribution to the determination of the analytes.

1. Instrument Startup

The following information guides instrument startup from the *Plasma Control Panel:*

1.1 Set the argon tank to 75 PSI.

1.2. Turn on the vent

1.3. Check tubing. It should not be flat, hard, worn, or discolored.

1.4 Check nebulizer for clogs (If necessary, clean nebulizer by blowing argon through the end. Do this through *Plasma Control, modifying levels* with nebulizer gas on)

1.5. If running samples using the autosampler, attach both tubing cartridges. The red tubing cartridge pumps rinse water to the autosampler (bottom position), while the orange tubing cartridge pumps sample to the instrument (top position). The tubing from the red cartridge should be in DI H_2O. If the instrument runs without sample or blank flowing through the nebulizer, it will automatically shut down.

1.6. Press F1 = startup

- If the argon was previously shutdown, then ignition may take a couple of attempts.
- Plasma startup default levels are as follows:
  Purge Time = 90
  RF Power = 950

1.7. Press F9 = continue

1.8. Press F2 = levels

- The default levels are as follows:
  Torch Gas: High Flow
  Auxiliary Gas: Medium 1.0 L/min
  Nebulizer Gas: ON (PSI) 28
  Approximate RF Power (W): 950
  Pump Rate (RPM): 130

1.9. Set the tension on the orange tubing cartridge by aspirating DI water and increasing the tension until the flow stops. Increase tension by one more notch. The pump must be off to perform this adjustment.

1.10. Check the sample nebulization. The flow should be even, not pulsing.

1.11. Press F9 = done keep

1.12. Press esc to return to the main menu. Allow 30 minutes for instrument warm-up.

2. Profile

After 30 minute warm-up, to profile the instrument choose *Setup* from the main menu and choose *profile* from the sub menu.

2.1. Aspirate a 5 ppm arsenic solution. It will take about 30 seconds for the solution to reach the torch.

2.2. Press F3 = automatic

- (Default settings for initial profile are as follows:)
  Flush time = 30 seconds
  Preintegration time = 0 seconds
  Integration time = 0.5 seconds

2.3. Press F1 = Run

2.4. If SS position is acceptable, hit done/keep (F9), if not,

2.5. Press F1 = Calc SS

2.6. Enter the vernier position (normally lists the previous position).

2.7. Press enter

2.8. Instrument will calculate the new vernier position. Set the instrument to this position.

2.9. Press esc (don't save), enter

2.10. Repeat the above steps until the peak offset is within $\pm$0.01. When acceptable, Hit F9 = done/keep and exit the screen.

2.11. Record information in Daily Optimization Record

3. Autosampler

To setup the autosampler table, choose *Operation* from the main menu, and choose *autosample setup* from the sub menu.

3.1. Enter autosampler table name, normally this will be "Full".

3.2. Press enter

3.3. If using an established table, Press F1 = Edit Set. Set the description, method name, etc...)

3.4. Press F1 = Edit Samples

3.5. Verify sample / standard queue
"S" means the instrument will standardize based on the indicated standards
"Q" means the instrument will run the indicated standard as a QCI
All "Q" flagged runs are from sample tubes located in the "L" rack (rack #1)

3.6. QC tables and sample tables can be used to flag data automatically. The use of these tables are built into the method.

3.7. Racks 2,3,4 are for samples and rack 1 is for QC.

3.8. The first sample is the procedure blank and the second is the LCS, when applicable. Enter all samples to be analyzed. Insert blanks and standards every 10 samples.

3.9. Verify that rack positions correspond with those in the table.

Note: Inserting QC is case sensitive. The QC label must match exactly otherwise it is treated as a different QC sample.

4. Queue

Calibration Standards

Transfer 1.0 mL QC 19 (100 mg/L for each element) and 100uL 1000ppm Ba stock solution and dilute to 100 mL. Label as Standard A. Final diluted standard shall contain 10% HNO_3 and 5% HCL, and 1.000 mg/L of the following elements:

As Ba Be Cd Cr Cu Fe Pb Mn Ni Se Co Sb Tl V Zn

Transfer 5.0mL ICSA, 0.25mL of 10,000ppm K & Na stock solutions and dilute to 100mL. Label as Standard B. Final diluted standard shall contain 10% HNO3 and 5% HCL

100 mg/L Fe
250 mg/L Al, Ca Mg
25 mg/L Na, K

Transfer 100uL 1000ppm Ag stock solution and dilute to 100 mL with 15% HCL

Calibration Blank - Deionized water containing 10% HNO3 and 5% HCL

Analyze the blank, STDA, STDB, & STDC (as required), in the uncalibrated mode. The instrument will measure the response for each element. Re-analyze the same standards after calibration. %R for each element following calibration is 95 - 105%.

Instrument Performance Check (IPC)

0.500 mg/L Ag
1.000 mg/L: As Ba Be Cd Cr Cu Fe Pb Mn Ni Se Co Sb Tl V Zn
250 mg/L Al, Ca Mg
25 mg/L Na, K

Prepare using the same technique and stock solutions as above, but dilute to 200mL

Instrument Calibration Verification Standard (ICV): second source standard

0.500 mg/L: As Ba Be Cd Cr Cu Fe Pb Mn Ni Se Co Sb Tl V Zn
50 mg/L Fe
125 mg/L Al, Ca Mg
1.25 mg/L Na, K

Analyze the ICV standards following calibration.

Low Standard Verification

Dilute STDA by 100 in deionized water containing 10% HNO3 and 5% HCL. This will provide a solution containing 0.010 mg/L of the analytes of interest. If needed, perform the same dilution of STDB and STDC. Analyze at the beginning and end of each analytical run to verify the low end of the linear range.

ICSA

10mL ICSA stock solution diluted with deionized water containing 10% HNO3 and 5% HCL to 100 mL.

500 mg/L Al Ca Mg, 200mg/L Fe

ICSAB

10mL ICSA stock solution, 1.0mL ICSB stock solution diluted with deionized water containing 10% HNO3 and 5% HCL to 100 mL.
500 mg/L Al Ca Mg
200 mg/L Fe
1.0 mg/L Ag Cd Ni Pb Zn,
0.500mg/L Ba Be Co Cr Cu Mn

Laboratory Reagent Blank (LRB) or Procedure Blank (PB)

De-ionized water processed through complete procedure, containing same acid concentration as all samples and standards. See prep procedure.

Laboratory Fortified Blank (LFB) or Laboratory Control Standard (LCS)

Spiked de-ionized water processed through complete procedure, containing same acid concentration as all samples and standards. See prep procedure.

Use ICV solutions for spiking:

0.02 mg/L Ag
0.200 mg/L all other analytes

Laboratory Fortified Matrix (LFM) or Matrix Spike (MS)

Use ICV solutions for spiking:

0.02 mg/L Ag
0.200 mg/L all other analytes

Reanalyze IPC and Calibration Blank after every ten sample and at the end.

QCS: Quarterly Performance Program - APG
After the preparation of stock or calibration standard solution

NOTE: All standards have a shelf life of one year from date of purchase or date of preparation.

5. Sample Analysis

To run samples choose *Operation* from the main menu, and choose *Analysis* from the sub menu.

5.1. Enter the method name, normally this will be "HSL".

5.2. Press F9 = autosampler

5.3. Enter table name, this will normally be "full".

5.4. Press F1 = run

5.5. The acidified rinse blank is aspirated between each sample. The concentration of acid in the rinse blank should match the concentration of acid in the calibration standards.

6. Shutdown

To shutdown the instrument choose *Setup* from the main menu and choose *Plasma Control Panel* from the sub menu.

6.1. Flush the sample tubing with DI water and then completely drain the tubing by aspirating air until the tubing is clear of sample.

6.2. Press F7 = Shutdown

6.3. Unhook tubing cartridges.

6.4. Shut off vent fan and argon.

7. Quality Control

Quality Control Indicator (QCI)	Frequency	Control Limit (Interim)	Control Limit (Statistical)
Initial Calibration	Daily	NA	NA
Calibration Blank	At the beginning and end, and after every ten samples	<Reporting Limit	
Calibration Check	Daily	±5%	NA
Initial Calibration Verification (ICV)	At the beginning of the batch	90-110%	NA
Interference Check Standard	At the beginning and end of run batch	80%-120%	NA
Laboratory Reagent Blank (LRB)	Per batch, where a batch is defined as daily or every 20 samples - whichever is less	<DL	NA
Laboratory Fortified Blank (LFB)	Per batch, where a batch is defined as daily or every 20 samples - whichever is less	90-110% R	NA
Duplicate	10%	+/- 20 RPD	NA
Laboratory Fortified Matrix (LFM)	10%	±25%	NA
Quality Control Sample (QCS) *	Quarterly - APG PE Program	±5% of the mean of three analyses	

A LFB and LRB are required only if the sample is digested. If samples are not digested, the Turbidity measurement must be documented as being <1 NTU.
* Also require after preparation of calibration standard solutions

8. Record Keeping

8.1. Record the percent recovery for each QCI on the raw data. Also record the RPD when appropriate.

8.2. QC data is filed by date, while sample data is filed chronologically by client.

8.3. Metals QC forms can be found in mother\winword\save\forms\metals

Approvals

Reviewed for Technical Accuracy by: William H. McHardy

Reviewed for Quality Assurance Compliance by: L. Franklin

Implementation Date: 4/16/98

End Use Date: ____________________